Phosphorous (Available) in Fertilizers: Direct Extraction Method 993.31

1.0 Scope

This method is suitable for the determination of percent direct available phosphorous (P_2O_5) in fertilizers. Both solid and liquid fertilizers can be analyzed by this method.

2.0 Summary

Fertilizer samples are extracted with ammonium citrate at pH 7.0 in the presence of disodium EDTA (ethylenediamine tetraacetic acid) to complex calcium and magnesium. The phosphorous is precipitated with quimociac reagent and the results are based on the weight of the precipitate.

3.0 Apparatus and Materials

3.1 Gooch crucible.

3.2 500 ml and 50 ml erlenmeyer flasks.

3.3 Oven.

3.4 Water shaker bath, heated.

3.5 Suction filtration apparatus.

3.6 Hot plate.

3.7 Glass fiber filter paper.

3.8 Fast flow filter paper. (Quantitative is suitable)

4.0 Reagents

4.1 Ammonium Citrate-EDTA solution: Dissolve 25 g disodium EDTA and 50 g dibasic ammonium citrate in 1.5 L H_2O. Nearly neutralize by adding 30 ml ammonium hydroxide, and then adjust pH to 7.00, with aqueous NH_4OH solution (1+1). Dilute to 2 L with H_2O. I use this but in a multiplication of five.

4.2 Nitric acid (1+1): Mix equal volumes of concentrated nitric acid and deionized water. Allow the solution to cool before using. Always add the acid to the water.

4.3 Quimociac Reagent: Dissolve 140 g of sodium molybdenum oxide dihydrate ($NaMoO_4.2(H_2O)$) in 300 ml of deionized water. Dissolve 120 g of citric acid in a mixture of 170 ml of concentrated nitric acid and 300 ml of deionized water and cool. Gradually add the molybdate solution to the citric acid-nitric acid mixture with stirring. Dissolve 10 ml of quinoline in a mixture of 70 ml of concentrated nitric acid and 200 ml of deionized water. Gradually add this solution to the molybdate-citric acid-nitric acid solution, mix and let stand for 24 hr. Filter, add 560 ml of acetone, dilute to 2 liters with deionized water and mix.

5.0 Procedure

5.1 Weigh 0.5 g sample into a 250 wide-mouth volumetric flask. (Record the sample weight to the nearest 0.0001 g.)

5.2 Fill the shaker bath with distilled water to a minimum of ½ inch above the top of the bottle clamps. Switch on the shaker bath heater.

5.3 Transfer 100 ml of the ammonium citrate-EDTA solution into the 250 ml volumetric flask containing the sample and place in the shaker bath. Shake samples for 1 hour at 65 ± 2^0 C and at 200 rpm.

5.4 Remove flasks from shaker bath and let cool. Bring to volume with deionized water and mix thoroughly.

5.5 Allow the sample solution to stand for at least two hours or preferably overnight.

5.6 Preheat the hot plate. Dry the gooch with a glass fiber filter paper in it at 250^0 C for 30 minutes. Cool the gooch and paper in a desiccator and weigh to the nearest tenth of a milligram (0.0001 g).

5.7 Pipet an aliquot of the sample solution containing $\leq$ 25 mg P_2O_5 (10 ml) into a 500 ml erlenmeyer flask.

5.8 Dilute the contents of the erlenmeyer flask to about 50 ml with deionized water.

5.9 Add 10 ml of the (1+1) nitric acid and boil gently for 10 minutes. Cool and dilute to about 150 ml.

5.10 Add 50 ml of quimociac reagent (using a 50 ml erlenmeyer flask) to the 500 ml erlenmeyer flask.

5.11 Invert the 50 ml erlenmeyer over the opening of the 500 ml erlenmeyer. Place on

the hot plate in a well-ventilated hood and boil one minute.

5.12 Cool the sample solution to room temperature, swirling carefully 3 to 4 times during the cooling.

5.13 Filter the solution into the gooch with a glass fiber filter paper previously dried at 250^0 and weighed to the nearest 0.0001 g.

5.14 Wash with three to five 25 ml portions of deionized water.

5.15 Dry the crucible and contents for 30 minutes at 250^0 C, cool in a desiccator to room temperature and weigh to the nearest tenth of a mg (0.0001 g).

5.16 Liquid fertilizers are analyzed by weighing directly into a 250 ml wide-mouth volumetric flask (record the sample weight) without filtering. Reagent blanks should be run with each new batch of reagents.

6.0 Calculations

6.1 Determine the reagent blank by subtracting the weight of the crucible and paper for the blank from the weight of the crucible, precipitate and paper for the blank.

6.2 Subtract the reagent blank from the weight of precipitate for each sample to obtain the net weight of precipitate.

6.3 Calculate % available P_2O_5 as follows:

$$\% \text{ Direct Available } P_2O_5 = \frac{(\text{net wt. of ppt.})(0.032074)(100)}{(\text{g sample})(\text{aliquot}/\text{dilution})}$$

Aliquot= Aliquot of combined extracts used for precipitation
Dilution= Total volume to which combined extracts were diluted.

7.0 Quality Control

7.1 Check pH of the ammonium citrate-EDTA.

7.1.1 Check and adjust if necessary upon initial preparation.

7.1.2 Check before use, adjust if necessary and document on work list.

7.2 Temperature of baths and ovens.

7.2.1 Check temperature of citrate shaker bath before use with a calibrated

thermometer and document on worklist if not 65 degrees C.

7.2.2 Check the temperature of the drying oven before use with a calibrated thermometer and document on worklist if not 250 degrees C.

7.3 Blanks.

7.3.1 Run at least 4 blanks on each new lot of ammonium citrate if you see an abnormally high result and the checks. The chemicals used today have less contamination than years ago.

7.3.2 Calculate an average and document.

7.4 Monitor time.

7.4.1 Water extraction should be completed in less than one hour.

7.4.2 Residue from water extract should be transferred to 65^0 ammonium citrate-EDTA solution within one hour of completion of water extraction.

7.4.3 Samples should be extracted in 65^0 C ammonium citrate-EDTA for one hour.

7.4.4 After taking aliquot for precipitation with quimociac reagent and adding 1+1 HNO_3, solution should be boiled for ten minutes.

7.4.5 After adding quimociac reagent, samples should be boiled for one minute.

7.4.6 Crucibles and precipitate should be dried for 30 minutes at 250^0 C.

*Note: The extraction time may take longer since the ammonium citrate-EDTA needs to be brought to temperature, not just the water.

8.0 Bibliography

Official Methods of Analysis (1995) 16th Ed., AOAC, Washington, D.C., sections 2.3.14, 2.3.13e, 2.3.03c.